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Tensile Strain Measurements of Ceramic Fibers Using Scanning Laser Acoustic Microscopy

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TENSILE STRAIN MEASUREMENTS OF CERAMIC FIBERS USING SCANNING LASER ACOUSTIC MICROSCOPY

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SUMMARY

A noncontacting technique using scanning laser acoustic microscopy for making in situ tensile strain measurements of small diameter fibers was implemented for the tensile strain analysis of individual Nicalon SiC fibers (nominal diameter 15 μm). Stress versus strain curves for the fibers were plotted from the experimental data. The mean elastic modulus of the fibers was determined to be 185.3 GPa. Similar measurements were made for Carborundum SiC fibers (nominal diameter 28 μm) and Saphikon sapphire fibers (nominal diameter 140 μm), yielding an elastic modulus of 401 and 466.8 GPa, respectively.

INTRODUCTION

Ceramic-ceramic composites have elevated temperature properties which make them attractive candidates for high performance aerospace structural applications. These materials have high specific strengths at elevated temperatures, high thermal stability, resistance to thermal shock and oxidation, and an estimated maximum operating temperature of 3000 °F. In addition, the ceramic reinforcing fibers enhance the composite system's resistance to fracture and catastrophic failure.

Accurate quantitative characterization of the reinforcing fibers is essential for the reliable characterization of composite structures under their operating conditions. However, material property measurements of small diameter fibers are difficult to achieve by traditional methods.

In this work, a noncontacting ultrasonic method using scanning laser acoustic microscopy (SLAM) for measuring tensile strain was applied to selected ceramic fibers. This technique proved to be a convenient and reliable approach that did not alter the mechanical response of the fibers during loading and strain measurement.

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THEORY

The operation and design of the scanning laser acoustic microscope (SLAM)¹ has been described in the literature in references 1 and 2. Fundamentally, a piezo-electric transducer transmits an acoustic wave through the ceramic fiber specimen. This wave causes periodic fluctuations within the specimen characteristic of the material structure. A laser beam scanned over the surface of the fiber is modulated as a function of these periodic fluctuations. In essence, the scanning laser beam is used to detect acoustic disturbances in the specimen. This acousto-optical coupling yields a diffraction limited resolution capability of the system, i.e.,

$$R = 1.22 \Lambda f/^\star \quad (1)$$

where Λ is the acoustic wavelength in the specimen and $f/^\star$ is the f -number of the detection system. This permits the investigation and analysis of materials having small dimensions (to 11 μm).

The SLAM system has been used primarily for defect detection and characterization (refs. 3 and 4). However, by analyzing the acousto-optical signal response from the specimen directly, quantitative material behavior measurements are possible.

The detected acousto-optical signal is added to an electronic phase reference signal to form a series of interference fringes characteristic of the internal structure of the specimen. As the specimen is stressed, the modulation of the scanning laser beam is altered. This is indicated as a shift of the interference fringes which is directly proportional to the magnitude of the strain in the specimen.²

EXPERIMENTAL PROCEDURE

The ceramic fibers studied were Nicalon β -SiC (15 μm diameter), Saphikon α -Al₂O₃ (140 μm diameter), and Carborundum α -SiC (28 μm diameter). The individual fiber specimens were epoxied to 0.0254 mm thick stainless steel shim stock tabs as shown schematically in figure 1. The tabs were machined to yield a 1-in. gauge length configuration. The tabs were configured such that they could be cut, after the assembly was positioned in the measurement system, for load transfer to the fiber. One end of the tab was secured to the SLAM stage. A calibrated static load was applied to the other end via a frictionless air bearing pulley assembly designed to minimize the losses due to frictional effects between the SLAM stage and the shim tab. The experimental set up is shown in figure 2.

A small drop of water was placed between the piezo-electric transducer and the specimen. The water served as an acoustic couplant which promoted effective acoustic transfer from the piezo-electric crystal to the specimen. The crystal operated at 100 MHz for all experiments described in this work. A mirrored coverslip was placed 0.0254 mm above the specimen to increase the reflectivity of the diffracted signal. The signal from the SLAM interference fringes at the detector plane was directed to a 100 MHz oscilloscope. The raster line at the location of the fiber was selected and the fringe shift was measured directly from the analog signal on the scope. The fringe shift yielded a measure of the tensile strain on the fiber.

¹ Sonoscan, Inc.

²A more detailed description of the measurement technique used is presently being prepared for a Ph.D. dissertation at the University of Dayton.

After the strain measurement was completed, each fiber was removed from the SLAM system. The diameter of each was optically measured by laser diffraction using the United Calibration Corporation measuring system.

The strains for 15 individual Nicalon fibers were measured at three different load levels (10, 15, and 20 g). The data were analyzed using a complete randomized block experimental design. The elastic modulus calculated at each measurement point formed a 3 by 15 experimental matrix. In this design, the fiber to fiber variation was normalized and the residual variance was used to estimate the measurement uncertainty and reliability of the individual measurement. For each fiber, the strain data were plotted on stress (obtained from the measured loads and fiber diameters) versus strain curves and the elastic modulus of the individual fibers was calculated by the method of linear regression. The results obtained were compared with the values reported in the literature.

Saphikon α - Al_2O_3 and Carborundum SiC fibers were measured using similar procedures. The stress versus strain curves plotted from the data obtained were used to calculate the elastic modulus of each fiber.

RESULTS AND DISCUSSION

Figures 3 to 5 present stress versus strain curves for each of the fibers studied. The error bars shown are propagated from the measurement uncertainties. This propagated measurement error is generally less than 5 percent of the absolute strain measurement. For the fifteen Nicalon fibers studied, the mean elastic modulus, calculated from a linear regression of the measured stress and strain, was 185.3 GPa (fig. 3). This compares with a value of 193 GPa reported in the literature in reference 5 (a mean deviation of 2.8 percent).

The elastic modulus of the Saphikon Al_2O_3 fiber was calculated from the stress versus strain curves to be 466.8 GPa (fig. 4). This is within 1 percent of the value reported in the literature for c-axis α - Al_2O_3 (468.8 GPa) (ref. 6). The values reported are for c-axis fibers only. In some instances, elastic modulus values as low as 420 GPa were measured. However, further investigations of these fibers showed that they were off-axis by as much as 25° .

The elastic modulus of the Carborundum SiC fibers was determined to be 401 GPa (fig. 5). This value is 4.4 percent below the theoretical elastic modulus for this material (ref. 7). Subsequent analysis of the Carborundum SiC fiber studied showed a high degree of internal porosity, accounting for the low measured elastic modulus (ref. 8). This analysis was confirmed by the manufacturer (ref. 9).

The complete randomized block analysis of the resultant data shows that there is a statistically significant variation in the measured elastic modulus of the Nicalon SiC from fiber to fiber. The standard deviation of the elastic modulus is 22 GPa or approximately 12 percent about the mean. Accurate accounting of these deviations in the modulus is an important step in the material characterization process. In this case, the variance is primarily attributed to subtle differences in the microstructure from fiber to fiber. Figure 6 shows SEM micrographs of two Nicalon fibers which exhibit different textures on their fracture surfaces.

The SLAM measurement technique used in this study can also perform strain measurements on short gauge length (to 0.25 in.) specimens. This suggests potential success for measurement of the in situ tensile strain and elastic modulus of ceramic whiskers as well as measurement of local in situ strain distributions in larger specimens.

CONCLUSIONS

This work describes the results of measuring the tensile strain and elastic modulus of small diameter ceramic fibers using scanning laser acoustic microscopy (SLAM). An analysis of these properties for three different ceramic fibers is presented. The size and brittle nature of these fibers has previously prevented an accurate assessment of these properties. The SLAM measurement technique used allows analysis of material specimens having small dimensions and short gauge lengths.

ACKNOWLEDGMENTS

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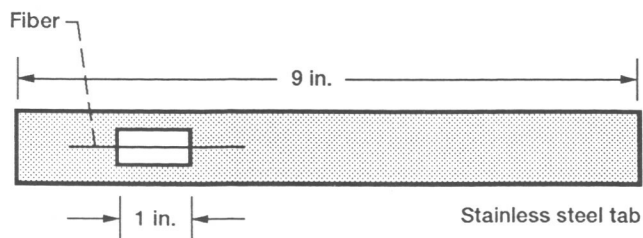


Figure 1.—Fiber specimen configuration.

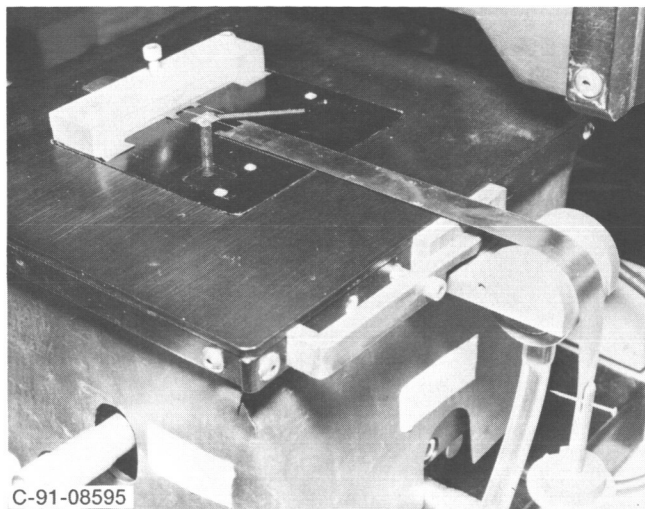


Figure 2.—Configuration for fiber strain measurement.

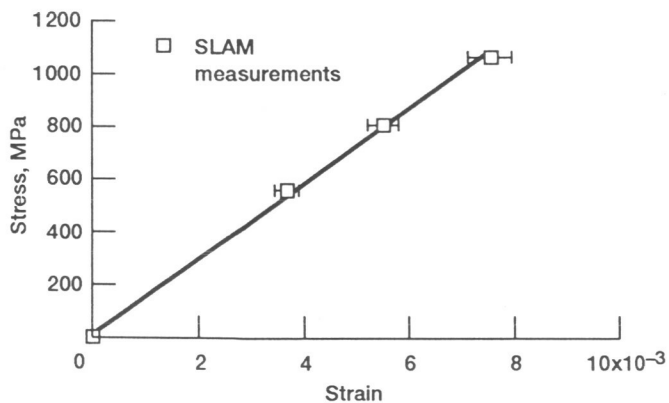


Figure 3.—Stress vs. strain for 15 μm Nicalon $\beta\text{-SiC}$ fiber.

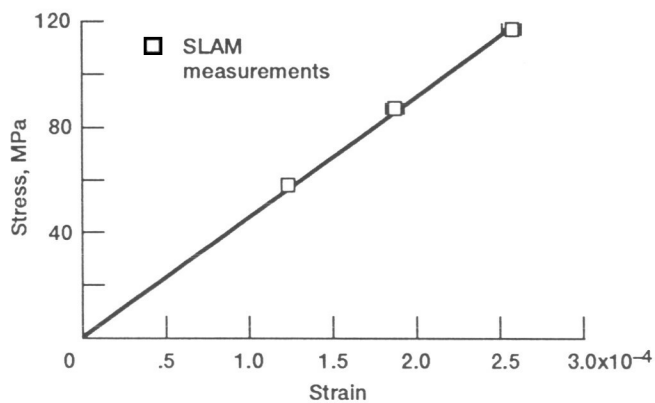


Figure 4.—Stress vs. strain for Saphikon $\alpha\text{-Al}_2\text{O}_3$ fiber.

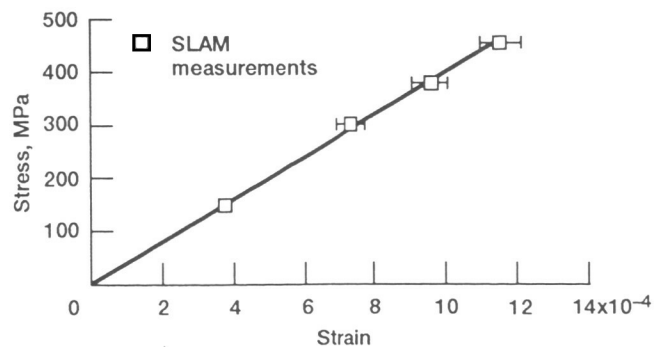


Figure 5.—Stress vs. strain for Carborundum $\alpha\text{-SiC}$ fiber.

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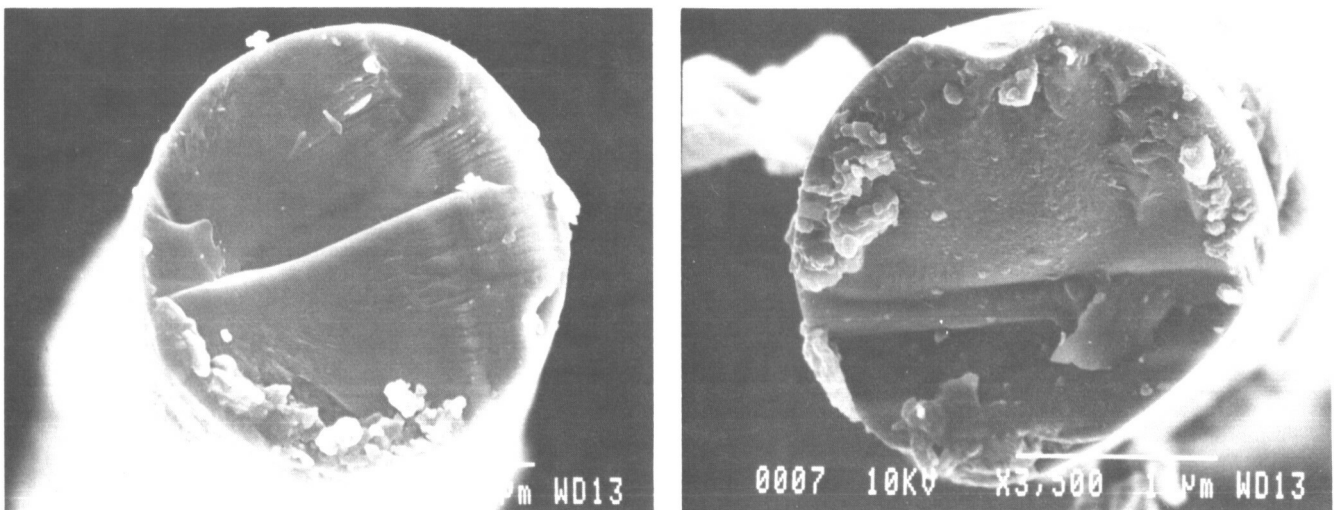


Figure 6.—SEM micrographs of Nicalon SiC fibers.

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